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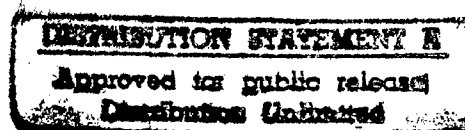
UNITED STATES ATOMIC ENERGY COMMISSION

**THE CREEP OF ZIRCONIUM IN WATER  
FROM 400° TO 600°F**

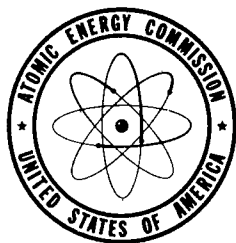
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Sylvania Electric Products, Inc.  
Bayside, New York



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THE CREEP OF ZIRCONIUM IN WATER FROM 400° TO 600°F

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ABSTRACT

2000-hour creep tests were made on 0.015-inch zirconium wire in water at temperatures ranging from 400° to 600°F. Values of secondary creep were determined and limiting creep stresses were obtained for creep rates of 0.1% and 1.0% in 1000 hours. Total elongation was measured after the test run of 2000 hours.

Work performed under Contract No. AT-30-1-Gen-366.

Sylvania Electric Products, Inc.  
Bayside, New York

## THE CREEP OF ZIRCONIUM IN WATER FROM 400° TO 600°F

By D. R. Brunstetter, N. P. Kling, and B. H. Alexander

### I. INTRODUCTION

Since zirconium offers possibilities as a constructional material, its mechanical properties at elevated temperatures are of great interest. The tensile properties of zirconium wire at elevated temperatures have been determined at this laboratory in a previous investigation<sup>(1)</sup>. The results are shown in the graphs in Fig. 1 and may be summarized as follows:

1. Tensile strength decreases from approximately 45,000 psi at 70°F to 10,000 psi at 1000°F.
2. Yield strength (0.2%) decreases from about 20,000 psi at 70°F to 9,000 psi at 1000°F.
3. Elongation decreases slightly from its room temperature value of 18% as testing temperature increases. In the range 800°F to 1000°F, the elongation increases to about 30%.

Creep tests in air at 25° and 200°C have also been made on 0.010 inch diameter zirconium wire in this laboratory<sup>(2)</sup>. Secondary creep rates are tabulated below:

25°C		200°C	
Stress (psi)	Secondary Creep Rate	Stress (psi)	Secondary Creep Rate
45,000	0.25% in 1000 hr.	33,000	2.0% in 1000 hr.
47,900	0.53	34,000	2.8
49,000	0.88	35,000	3.6
49,900	1.00	36,000	4.8
50,900	1.58	37,000	6.8
51,800	1.80	38,000	30.
52,700	8.46		
53,500	14.5		

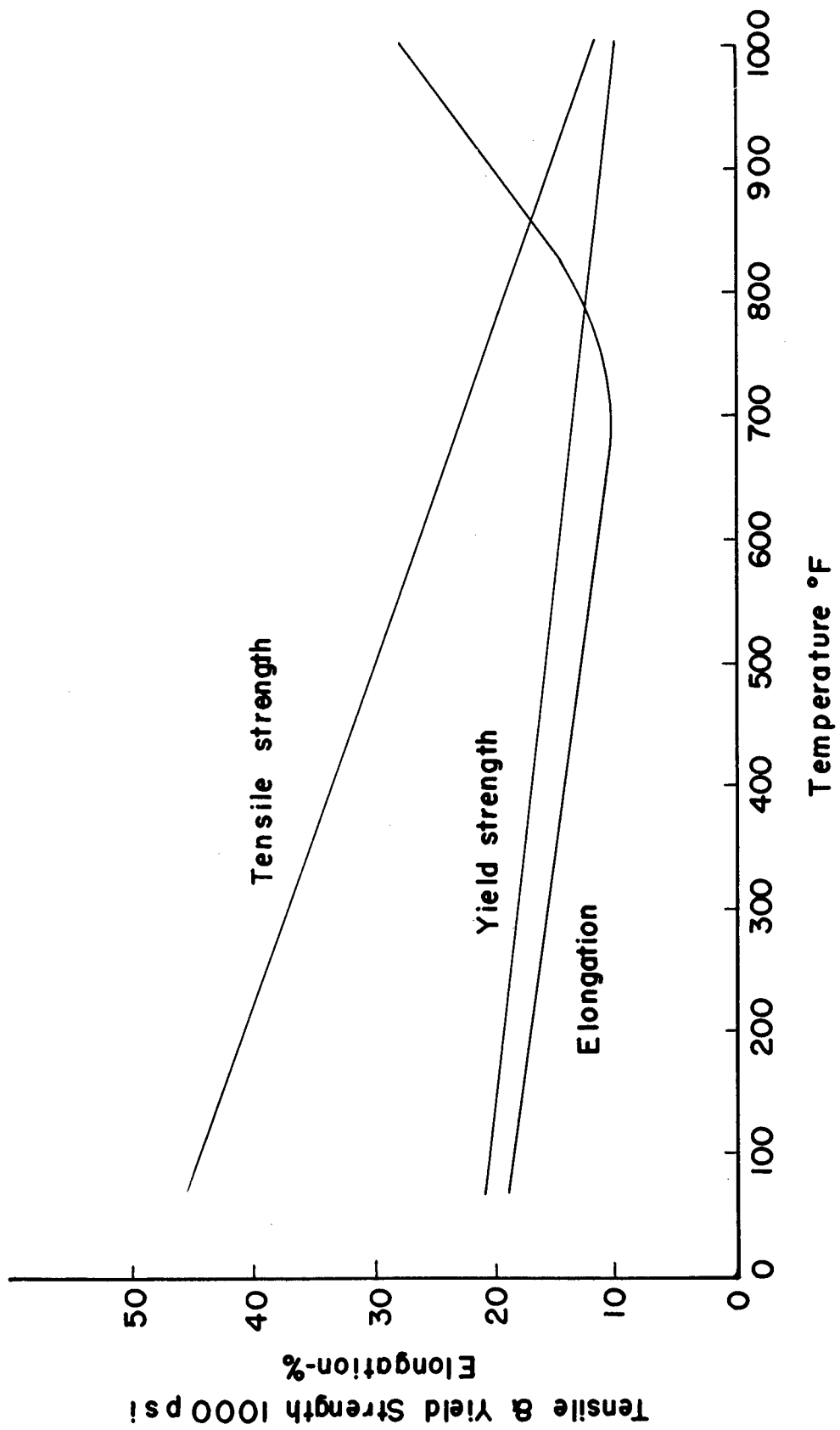


Fig.1

## I. INTRODUCTION (cont'd)

The material used in this investigation was of higher strength than that used for the elevated temperature tensile tests; its properties at room temperature were:

Tensile strength	-	70,000 psi
Yield strength	-	38,000 psi
Elongation	-	16%

## II. EXPERIMENTAL

Since pressures in the neighborhood of 1500 psi are necessary to maintain water in the liquid state at 600°F, a high-pressure autoclave was designed in which to conduct the tests. A sketch of the apparatus is shown in Fig. 2. The pressure vessel proper and the heating jacket are standard items from the American Instrument Company's line of Superpressure equipment. The sight fitting was obtained from Fischer and Porter Company; it was originally designed as the outer shell for a liquid level gage or flowmeter.

The pressure vessel consists of a stainless (Type 316) steel autoclave, 4-3/8" O.D. x 2-1/4" I.D. fitted with a stainless steel head. The head is clamped to the autoclave by means of a compression closure which screws on the outside of the autoclave; the head is forced into intimate contact with the autoclave by means of bolts passing through the compression closure and bearing on the head. A stainless steel ring gasket makes the joint between head and shell pressure tight. The upper wire grip, of stainless steel, is screwed to the head. The wire specimen, fastened to the upper wire grip, suspends the lower wire grip, weight bucket, grip extension, and sight point, all of stainless steel. The grip extension and sight point pass through a stainless steel tube screwed into the lower end of the pressure vessel and into the upper end of the sight fitting. The sight fitting has opposite windows of 3/4" thick pyrex glass, through which the movement of the sight point could be observed with a cathetometer.

The specimens, of 0.015 inch diameter wire, were gripped in sleeves of copper tubing approximately 0.080" O.D. x 0.020" I.D.; these were crimped on the ends of the specimens, and in turn were clamped into 3/32" diameter holes in the grip by means of set screws.

The interior temperature of each of the six pressure vessels was controlled by a Model C Micromax controller; the controlling thermocouple was placed between the heating jacket and pressure vessel. Another thermocouple was placed in the thermocouple well

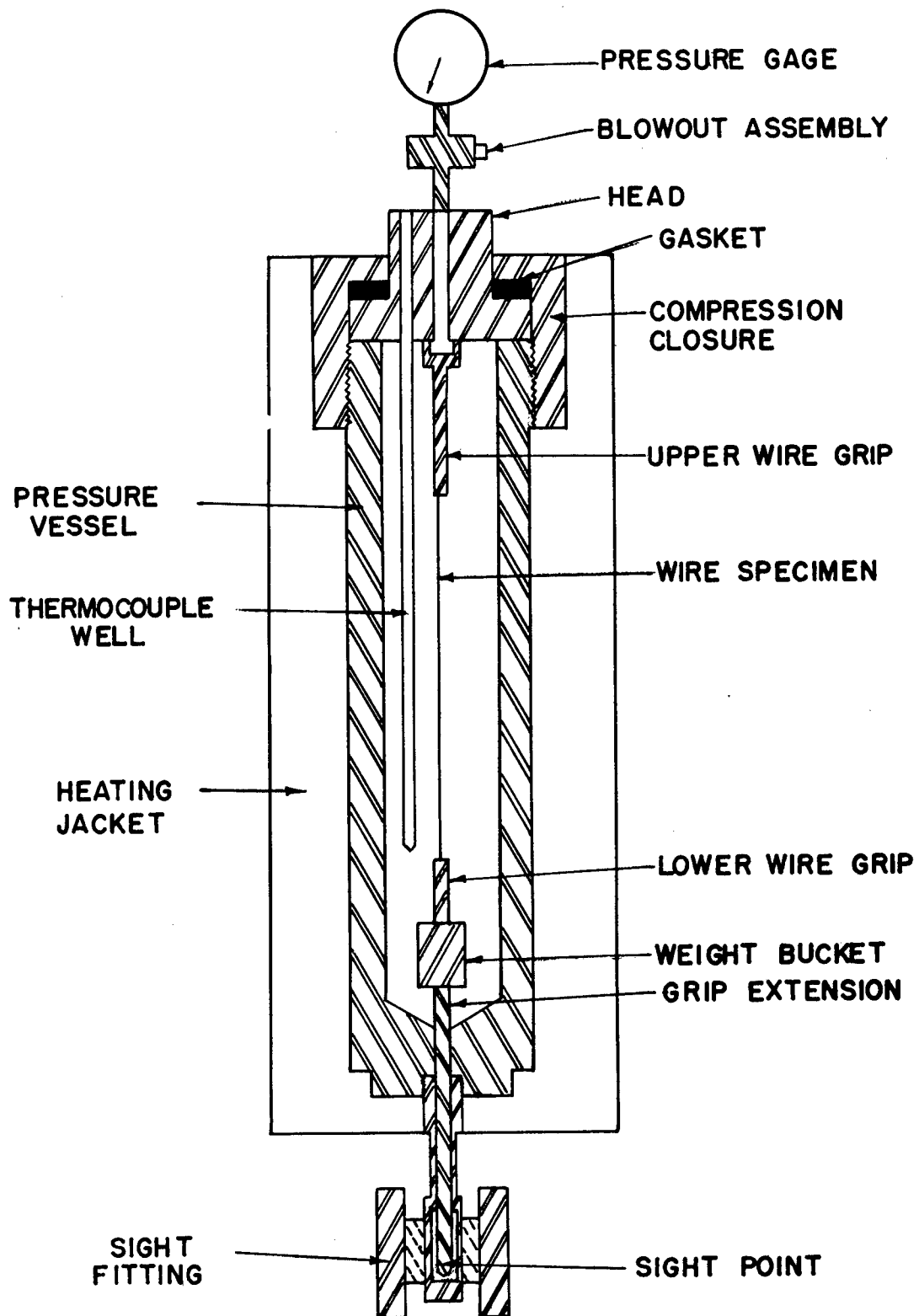


Figure 2

## II. EXPERIMENTAL (cont'd)

adjacent to the center of the wire specimen. These temperatures were recorded on a six-point Model S Micromax recorder. Temperature variation was approximately  $\pm 5^{\circ}\text{F}$  along the length of the specimen and the same with respect to time.

The wire specimen was 10 inches long; a cathetometer reading to 0.005 cm was used to read elongation. By this means, a percentage elongation of 0.02% could be detected.

The tests ran for 2000 hours. At the end of each test, the specimens were removed and examined for any evidence of corrosion; in no case was any corrosion detected.

The material tested was 0.015 inch diameter zirconium wire produced from iodide crystal bar by the Foote Mineral Company (Lot #911-11). Its processing history was as follows:

1. Crystal bar cold rolled to 0.125 inch
2. Vacuum annealed at  $1250^{\circ}\text{F}$  for 1 minute
3. Cold swaged through 0.125 inch die
4. Sheathed in brass
5. Swaged to 0.086 inch
6. Sheathed wire drawn to 0.0253 inch
7. Brass sheath removed in  $\text{HNO}_3$
8. Vacuum annealed on spools at  $1300^{\circ}\text{F}$  for 45 minutes  
(Grain size approximately 0.01 mm).

Room temperature tensile properties of this material are (average of three values):

Yield Strength (0.2%)	- 28,000 psi
Tensile Strength	- 53,000 psi
Elongation (4" gage)	- 21%

Calculations of loads necessary for the stresses used were all corrected for the buoyancy effect of the water in the pressure vessels, due regard being taken for the change in density of liquid water with temperature. In operation, the pressure vessels were filled with a volume of distilled water which, at the testing temperature, would fill the autoclave to within one inch of the top. When an interior temperature of  $250^{\circ}\text{F}$  was reached, pressure was relieved so as to bleed all air from the vessel, resulting in an atmosphere of liquid water surround-



## II. EXPERIMENTAL (cont'd)

ing the specimen. The vessels were operated at the following pressures:

<u>Temperature</u>	<u>Gage Pressure</u>
400°F	232 psi
500°	666
600°	1528

## III. DISCUSSION OF RESULTS

Curves of elongation versus time for the three temperatures investigated are shown in Figs. 3, 4, and 5. It should be noted at once that these curves include only a portion of the initial part of the creep curve. Since loading the specimens in the pressure vessels and sealing the vessels was a task that occupied most of a day, the specimens were subjected to loads overnight at room temperature. In addition to this, expansion of the pressure vessel itself during the time that it was being brought up to temperature (about six hours for 600°F) leads to further inaccuracies in the initial portion of the creep curves. These circumstances, however, do not affect the accuracy of the creep curves beyond the first day of testing, nor do they affect the values of secondary creep rate which we have obtained.

Values of secondary creep rate have been calculated from the straight line portions of the elongation-time curves. These are tabulated below:

400°F		500°F	
Stress	Secondary Creep Rate	Stress	Secondary Creep Rate
22,000 psi	0.11% in 1000 hr.	16,000 psi	0.10% in 1000 hr.
24,000	0.17	18,000	0.38
26,000	0.35	20,000	0.66
28,000	0.74	22,000	1.74
30,000	1.32	24,000	3.96

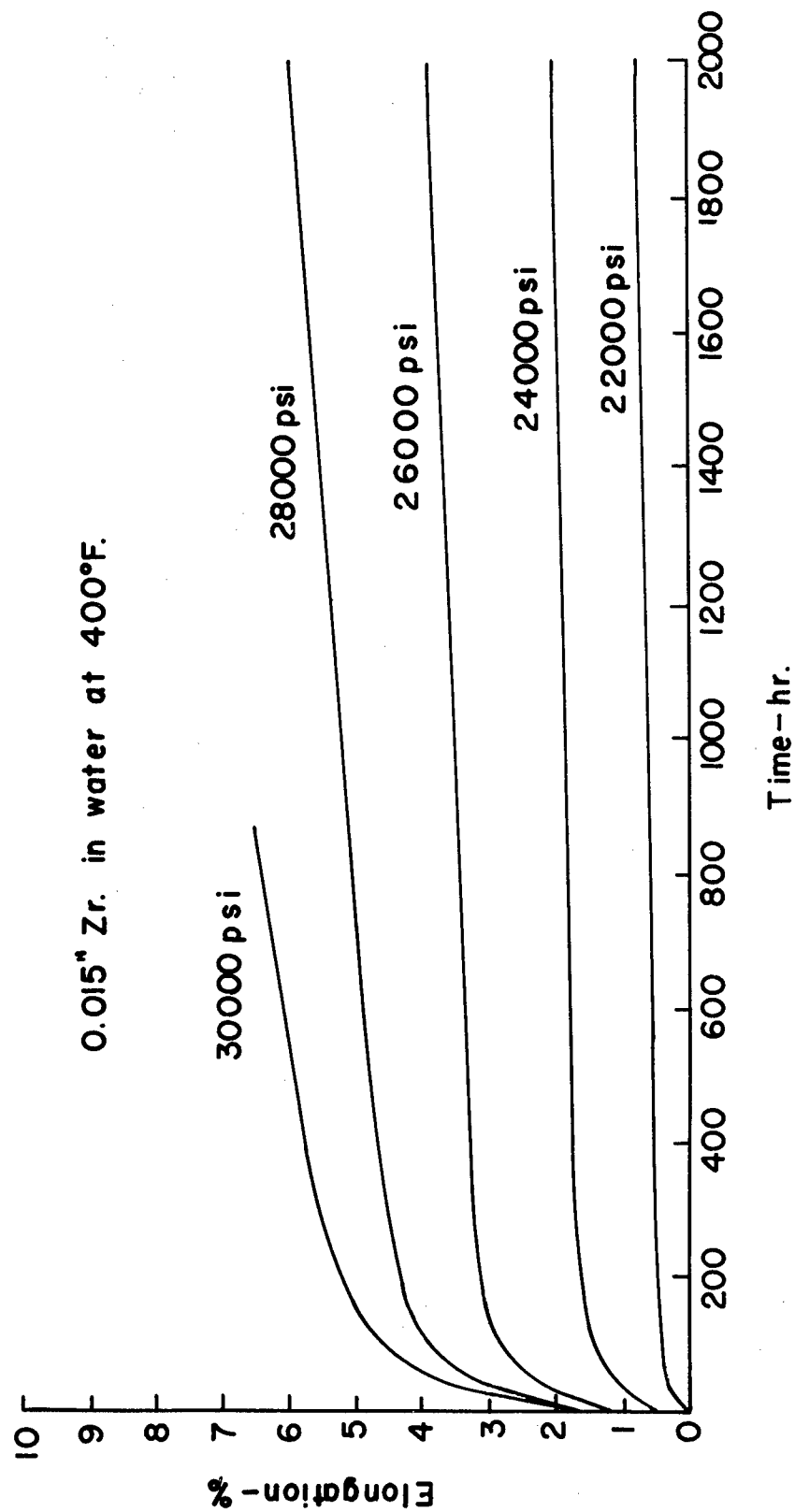


Fig. 3

0.015" Zr in water at 500°F.

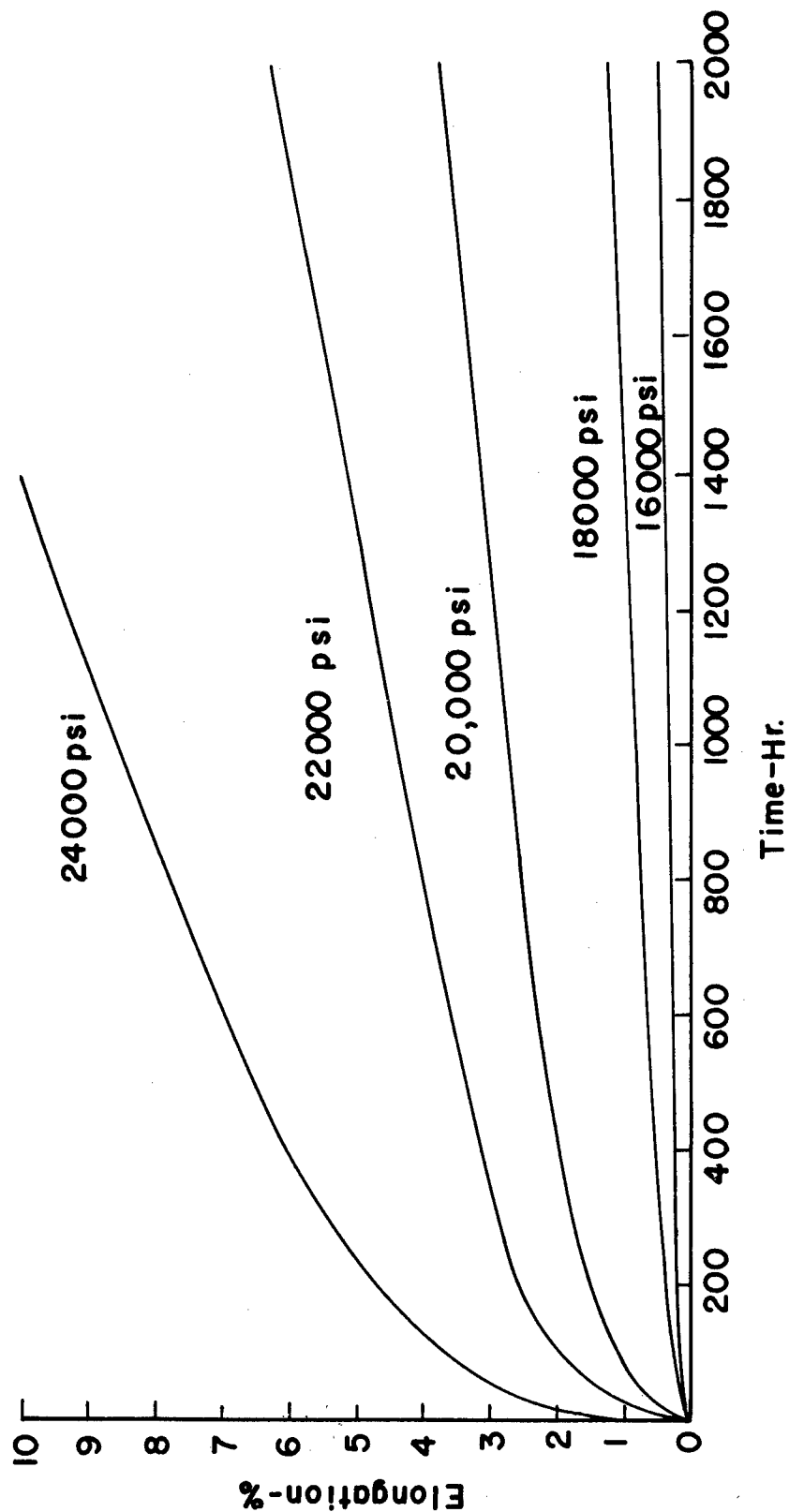


Fig. 4

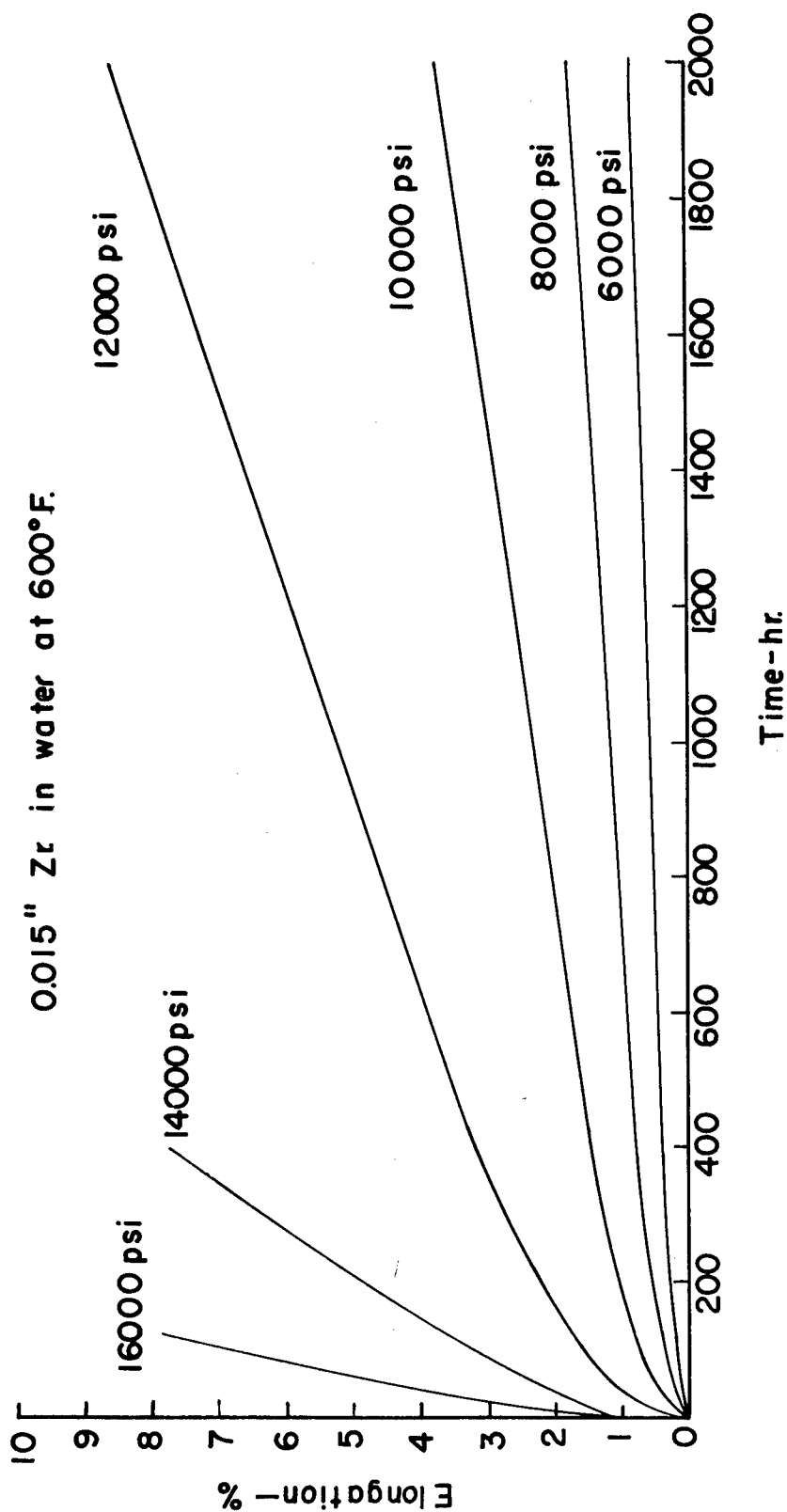


Fig. 5

### III. DISCUSSION OF RESULTS (cont'd)

600°F	
Stress	Secondary Creep Rate
6,000 psi	0.15% in 1000 hr.
8,000	0.64
10,000	1.51
12,000	3.35
14,000	14.1

These values are plotted in Fig. 6, as log creep rate versus stress, together with the data on 0.010 inch diameter wire given in the introduction. By extrapolation of the curves where necessary, limiting creep stresses for creep rates of 0.1% and 1.0% in 1000 hours may be obtained; these are tabulated below:

Temperature	Limiting Creep Stress for Creep Rate in 1000 hrs. for	
	0.1%	1.0%
400°F	22,000 psi	29,000 psi
500°	16,000	21,000
600°	5,000	9,000

A plot of limiting creep stress versus test temperature is shown in Fig. 7.

Specimens were measured following the test run of 2000 hours in order to determine the total elongation during the test. Results for the lowest stresses at each temperature are listed below, together with the secondary creep rate for that stress:

Temperature	Stress	Secondary Creep Rate	Total Elongation in 2000 hrs.
400°F	22,000 psi	0.11% in 1000 hr.	1.2%
500°	16,000	0.10%	1.5%
600°	6,000	0.15%	2.8%

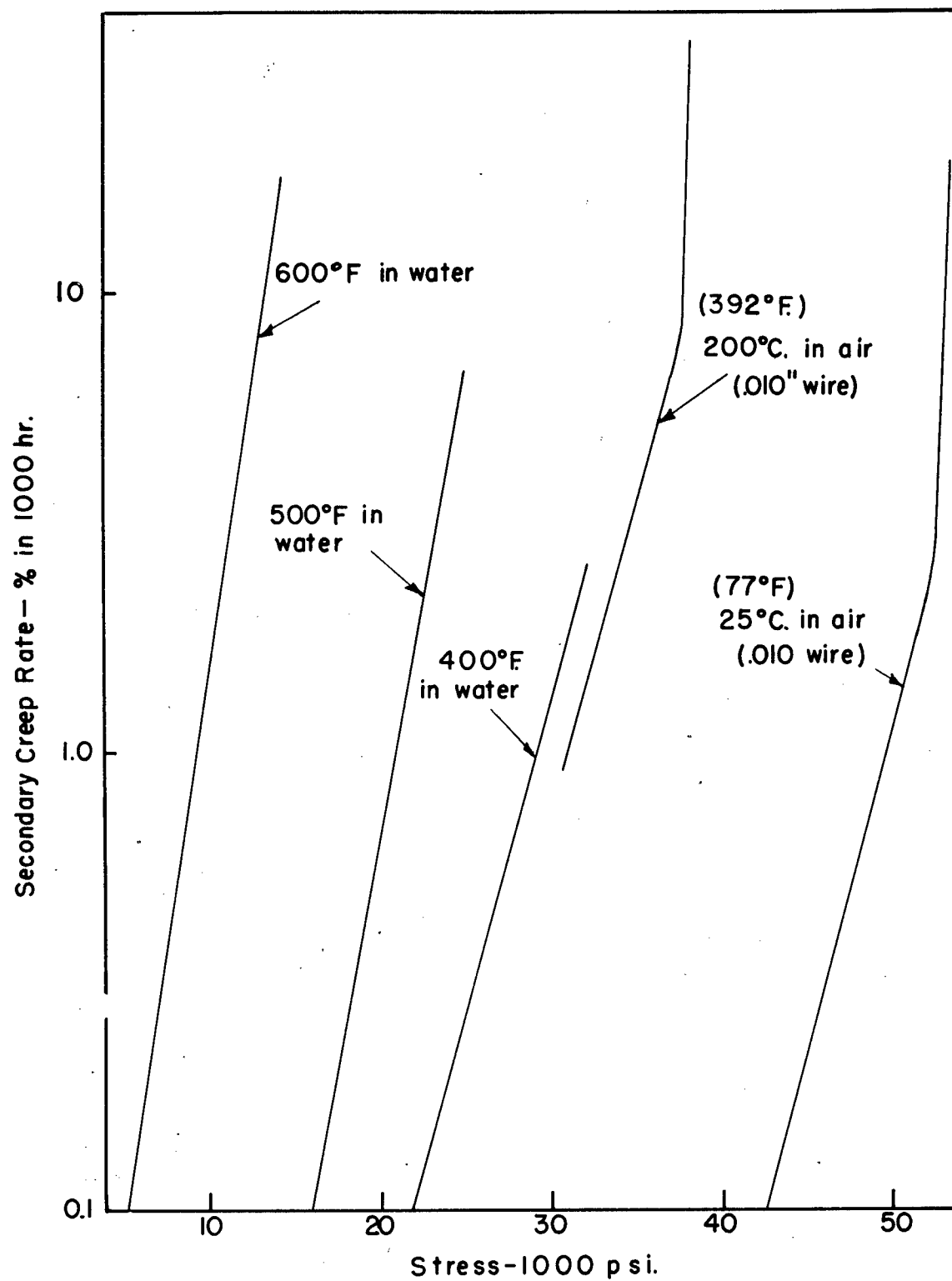


Fig. 6

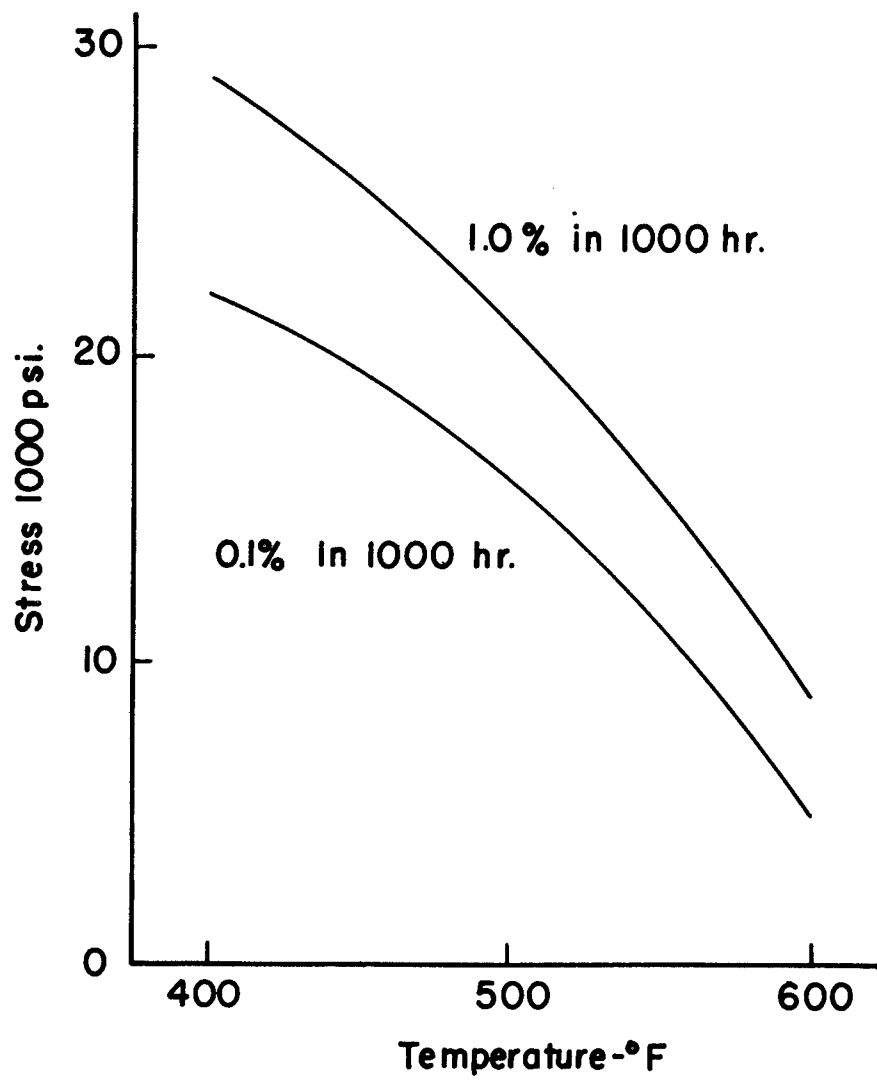


Figure 7

### III. DISCUSSION OF RESULTS (cont'd)

These data indicate that design stresses based on a knowledge of the secondary or minimum creep rate alone would be open to question as to their validity. In most cases, a total elongation of 1 to 2% could not be tolerated in an engineering design, even though the creep rate might be of the order of 0.1% in 1000 hours.

### IV. CONCLUSIONS

Creep tests of 2000 hours duration have been made on 0.015 inch diameter zirconium wire in water at 400°, 500° and 600°F. Secondary creep rates have been determined and limiting creep stresses calculated for creep rates of 0.1% and 1.0% in 1000 hours. These are listed as follows:

Temperature	Limiting Creep Stress for Creep Rate in 1000 hours of	
	0.1%	1.0%
400°F	22,000 psi	29,000 psi
500°	16,000	21,000
600°	5,000	9,000

It was found that stresses which caused secondary creep rates of about 0.1% in 1000 hours resulted in total elongation in 2000 hours of 1% to 2%. Such elongations make the use of design stresses based on secondary creep rates alone of questionable value.

### REFERENCES

1. Brunstetter, Kling and Alexander, "The Tensile Properties of Zirconium at Elevated Temperatures", NYO-1126, April 4, 1950.
2. Brunstetter and Alexander, "The Creep of Zirconium", SEP-17, June 9, 1949.